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PAPER

Analysis of corrosion rate, inhibition efficiency, and economic cost of XD3 reinforced concrete related to inhibitor and plasticiser types

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Abstract

The main problem in concrete structure is rebar corrosion, which is caused by the penetration and diffusion of chloride ions into reinforced concrete structures; therefore, it is essential to give the most attention to preventing or limiting the impact of this condition. So, in order to find a practical solution to the problem of corrosion of rebar, the current article focuses on demonstrating the beneficial effects of green and calcium nitrate inhibitors on chloride-induced corrosion. Thus, this paper presented an analytical procedure based on corrosion rates and the economic cost to evaluate the efficacy of such an inhibitor. Ten samples with various concrete mixtures were divided into two groups according to the type of plasticiser and inhibitor have been immersed in an aqueous solution of 3.5 wt% sodium chloride at room temperature for 18 months. The optimal results in which the corrosion rate was significantly reduced were for the two groups of concrete mixtures, first group was (C2), which contains 3 wt% orange peels extract inhibitor mixed with 2.4 wt% Oxydtron as water-resisting admixture, and the second group was (E2) included 3 wt% calcium nitrate inhibitor with 2.4 wt% Oxydtron as water-resisting admixture. These two groups samples have been showed more excellent corrosion resistance against the saline solution after the completed immersion period. The inhibition efficiency analysis showed that there is a direct relationship between the inhibitor concentration and the efficiency of corrosion inhibition. However, the inhibition efficiency in the case of samples containing calcium nitrate inhibitor was higher than their counterparts containing green inhibitor extract. Finally, the economic cost analysis showed that the cost of the green inhibitor extract-containing samples was lower than that of the calcium nitrate inhibitor-containing samples.

1. Introduction

In addition to its considerable safety hazards, reinforced concrete damage is one of the reasons for the corrosion of the steel reinforcement, which generates large economic losses. In urban areas, carbonation can occur as a result of chloride ions in the marine zone, which can also cause fast deterioration or the use of thawing salts [1–5]. Because of the high alkalinity of the concrete pore solution, high-quality concrete generally offers great protection for steel reinforcing, allowing the rebar to be in a passive condition for extended periods of time before corrosion starts. Corrosion can be started by either a drop in alkalinity brought on by carbonation or by the loss of the passive layer by chloride ion assault. Concrete porosity and exposure class are additional crucial variables in rebar corrosion [6–11]. To melt snow or stop it from building up on roads and bridges, several countries, including the United States, Canada, and Europe, use salt. Although this technology has advantages, it has a detrimental impact on steel structures because chloride ions attack steel and gradually remove the passive protective layer of steel, which causes local corrosion [12–19]. According to figure 1, an electrochemical mechanism accounts for how well steel reinforcement resists corrosion. This process is activated when corrosive ions in the concrete structure are present in changing quantities. This results in the formation of potential

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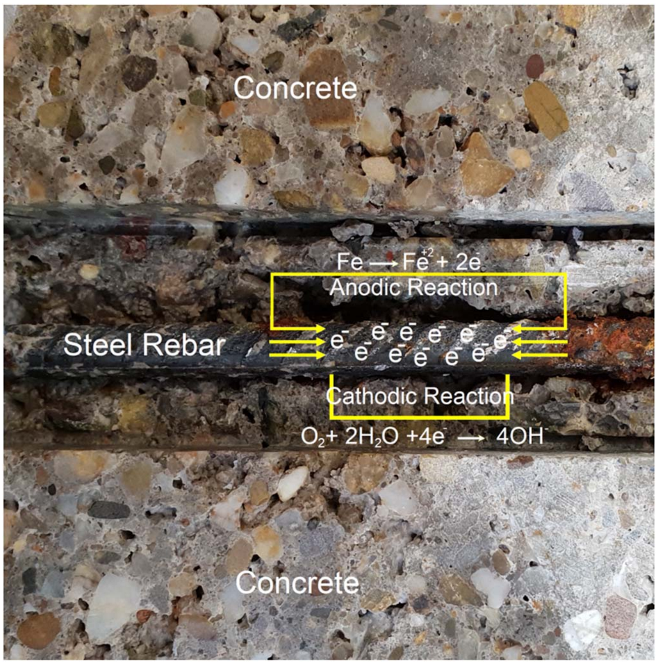
Concrete
$Fe \longrightarrow Fe^{+2} + 2e^{-}$
Anodic Reaction
Steel Rebar
e^{-}
Cathodic Reaction
$O_2 + 2H_2O + 4e^{-} \longrightarrow 4OH^{-}$
Concrete

Figure 1. Illustration of the corrosion procedure.

corrosion cells and electrochemical cells, which are characterised by electron and ion movement between the cathodic and anodic regions [1, 20–22]. Corrosion-resistant steels, cathodic protection, fusion-bonded epoxy coatings, corrosion inhibitors, and high-performance admixtures are some methods for reducing steel corrosion. Corrosion inhibitors, the most popular of these techniques, are easy to apply and very affordable. Inhibitors prevent corrosion by increasing the pH of the concrete or eliminating dangerous ions that might corrode steel [23–25].

When the inhibitor ions or molecules are absorbed by the reinforcing surface, the inhibitors usually form a hydrophobic coating. It lessens corrosion on reinforcement by inhibiting cathodic or anodic reactions. Utilising inhibitors with strong inhibitory effects is easy and inexpensive [26–29]. The development of 'green inhibitors,' as they are frequently referred to, has been promoted in the context of corrosion inhibitors due to the environmental issues that are caused by most chemicals in general and those used to prevent corrosion in particular. Green inhibitors are chemicals and chemical processes that strive to lessen or eliminate negative environmental consequences, according to the US Environmental Protection Agency's guidelines. Less waste, non-toxic products, and greater performance may be involved when these substances are utilised and processed. This kind of corrosion inhibitor is referred to as environmentally friendly inhibitors [30–36]. Plant extracts include a wide range of chemical molecules. The natural components of these extracts include heterocyclic macromolecules that contain nitrogen, oxygen, and sulphur and can form protective layers to block the entry of harmful ions like Cl^{-}, $SO4^{-2}$, and CO_2 and remove water molecules. These extracts also form a protective interface between the harmful ions and the steel surface through diffusion and are adsorbed at the steel-concrete interface through secondary molecular interaction with metal and metal oxide [37–42]. Another group [43] used compounds such as carboxylates, which can be recognised by the presence of the carboxylic acid (-COOH) group, as organic inhibitors. These compounds, like amines, have the ability to bind to the surface of metals to produce an organic coating layer that shields steel from corrosion. An increase in setting time and a lowering of the resistance rate increment are two adverse effects of carboxylate-based inhibitors in concrete that result in a decreased cement hydration rate [44–46]. Inorganic and organic inhibitors are frequently used as corrosion inhibitors to take on the role of the well-liked calcium nitrite inhibitor, which rose to prominence in the late 1980s until European regulations started to prohibit its use due to safety and environmental concerns. Inhibitors, both chemical and inorganic, have been studied as alternatives to these problems. The need to introduce a non-toxic, environmentally friendly inhibitor into concrete constructions to stop the phenomenon of rebar corrosion served as the impetus for the study of nitrate-based compounds [47–49]. The main goal of this paper is to present an experimental study that was conducted on observing and evaluating the corrosion processes that occurred in steel bar-reinforced concrete samples by analysing the corrosion rate, inhibition efficiency, and economic cost of XD3-reinforced concrete related to inhibitor and plasticiser types.

Table 1. Quantities of materials used and their cost.

	Material	Quantity, kg/m^3	Price, USD/kg
Cement		400	0.11
Additive	Sand (60%)	1089	0.14
	Gravel (40%)	726	0.14
Plasticisers	Super-plasticiser	2.4	1.77
	Water-resisting admixture	2.4	0.20
Inhibitors	Green inhibitor	4 (1 wt%), 12 (3 wt%)	0
	Inorganic inhibitor	4 (1 wt%), 12 (3 wt%)	0.49
Water (w/c = 0.43 planned/targeted value)		172	1.41

Table 2. The approximate chemical composition of steel rebar.

Element	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Fe
Concentration, wt%	0.19	0.78	0.24	0.027	0.035	0.11	0.11	0.27	0.02	98.22

2. Materials and methods

2.1. Materials

Table 1 shows the details of the materials used and their quantities as follows:

1. Cement: type Portland slag cement CEM II/A-S 42.5 R.
2. Aggregates: which include fine sand (0–4 mm) and coarse gravel (4–8 mm) according to EN 12620 [50].
3. Inhibitors: Orange peel extract as a green inhibitor, and calcium nitrate: as an inorganic inhibitor.
4. Plasticisers: Mapei Dynamon SR 31 as a super-plasticiser admixture, and Oxydtron as a water-resisting admixture.
5. Tap water: for preparation of the cement mixture and samples curing process.
6. Steel rebar: The rebar has a cross-section of 8 mm in diameter and 10 mm in length. The elementary chemical components and chemical composition of steel rebar were detected by using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), and the results of this analysis are listed in table 2.

2.2. Cost calculations

Cost calculations (purchase cost only, without manufacturing cost) of the materials used were taken into account in order to compare the cost of the natural botanical inhibitor represented by orange peels and the traditional inhibitor, which is calcium nitrate. Material prices are listed in table 1.

2.3. Orange peels extraction procedure

The orange peels were used to extract the green inhibitor solution. The extraction was carried out using the Soxhlet extraction process at BorsodChem Zrt., Hungary. The extracts from the Soxhlet apparatus were rotary-evaporated to expel the methanol by using vacuum evaporation at 40 °C and 60 mbar pressure.

2.4. Samples preparation

As shown in figure 2 and depending on EN 197-1 and EN 12620 European standards [50, 51], ten concrete mixtures with two different plasticisers and corrosion inhibitors were used to produce 7 × 7 × 7 cm cubic samples. Based on the European mix design method (XD3 class), concrete mixes were made to have a compressive strength of C35/45 after 28 days.

The practical procedure for the preparation of concrete samples is shown in figure 3. In the beginning, the initial mixing of the basic materials and additives was done manually in two separate mixtures to ensure complete homogeneity between these materials. The plasticisers have been added in a fixed weight percentage to all ten mixtures, which is 2.4 wt%. The initial mixing also includes mixing the inhibitors with water (each inhibitor individually) according to the specified weight percentages (1 wt% and 3 wt%) to ensure that the inhibitors are properly disseminated and distributed uniformly throughout the cement mixture thereafter. The

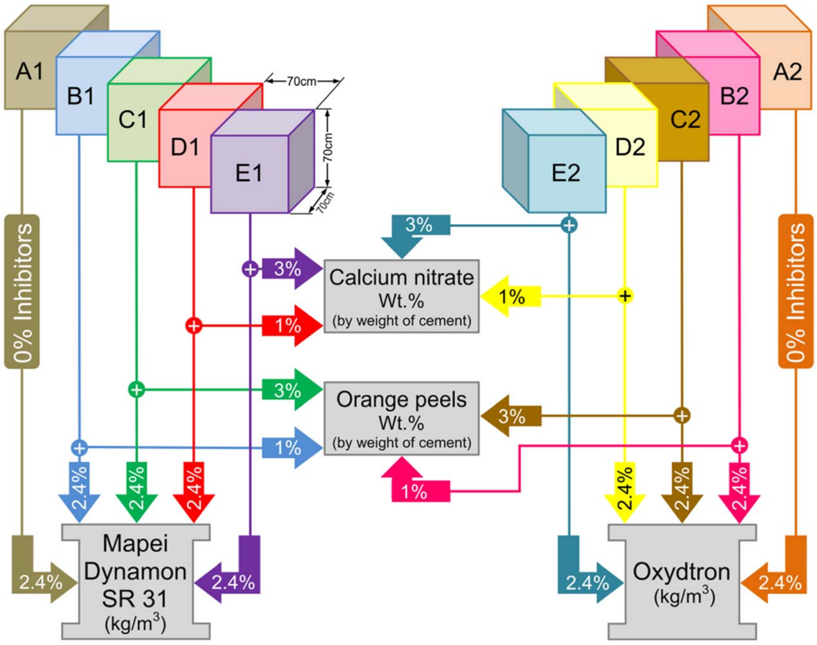

Figure 2. Concrete mixtures ingredients.

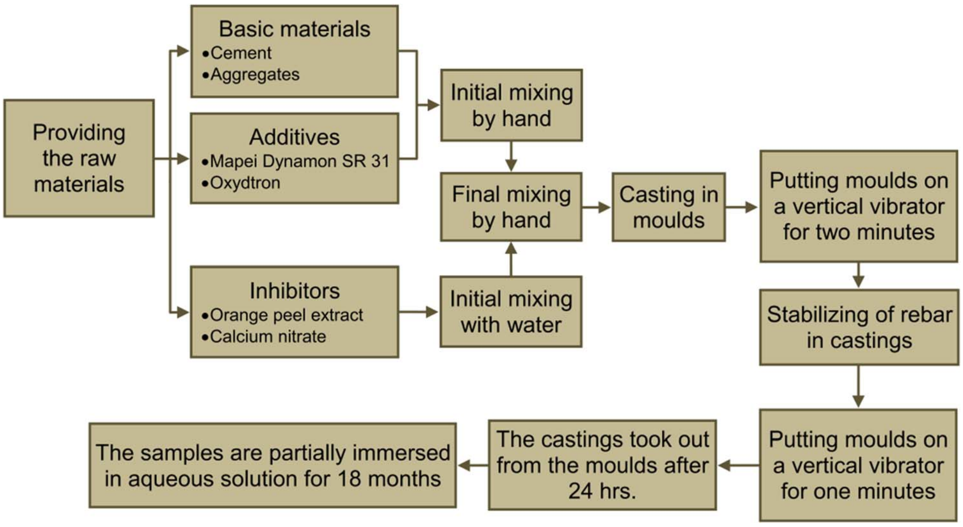
Providing the raw materials
Basic materials
•Cement
•Aggregates
Additives
•Mapei Dynamon SR 31
•Oxydtron
Inhibitors
•Orange peel extract
•Calcium nitrate
Initial mixing by hand
Final mixing by hand
Initial mixing with water
Casting in moulds
Putting moulds on a vertical vibrator for two minutes
Stabilizing of rebar in castings
Putting moulds on a vertical vibrator for one minutes
The castings took out from the moulds after 24 hrs.
The samples are partially immersed in aqueous solution for 18 months

Figure 3. Procedure of samples preparation.

final mixing is performed by adding water-containing inhibitors to the cement mixture created by the initial mixing, as well as physically mixing until full mixing is produced. The concrete mixture is poured into cube-shaped wooden moulds with the dimensions shown above. After the casting process is completed, the moulds are placed on a vertical vibrating machine for three minutes, including two minutes of initial vibration, to ensure the removal of bubbles from the mixture, the homogeneity of their distribution in the mould, and the filling of voids. A minute of secondary vibration is carried out after fixing the steel rebar in the middle of the mixture, and it aims to fill the voids that may have formed after the rebar implantation process and ensure that the rebar is surrounded by the mixture. To keep the rebar fixed in its position and not move down when the mould is subjected to vibration, it is attached to a plastic cover with a perforation in the middle that is the same size as the rebar and the dimensions of the mould. The concrete mixture is left inside the moulds for 24 h at a temperature of 25 °C in order to complete the hardening process. Following the completion of the hardening process, the samples were removed from the moulds and partially immersed in a 3.5 wt% NaCl solution for 18 months. The samples that have been produced and shown in figure 4 will be appropriate for electrochemical polarisation measurements.

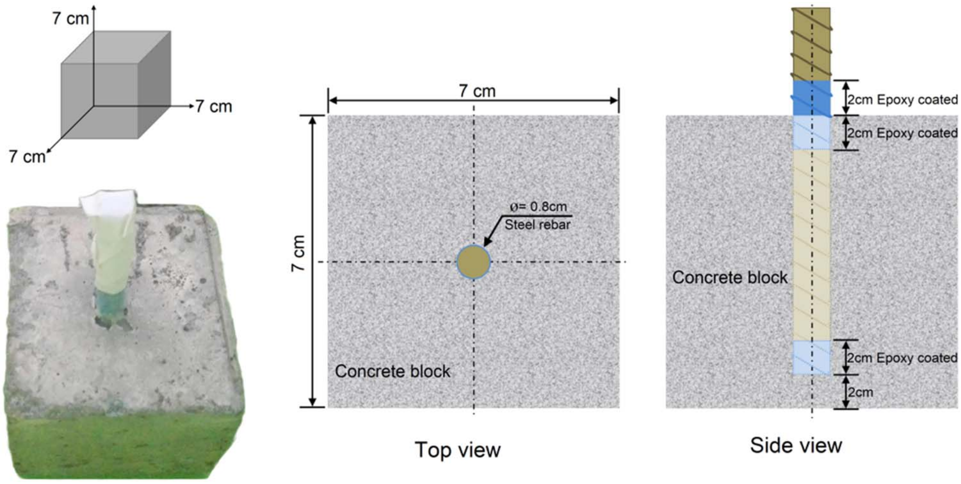

Figure 4. Schematic illustration for electrochemical polarisation measurements sample.

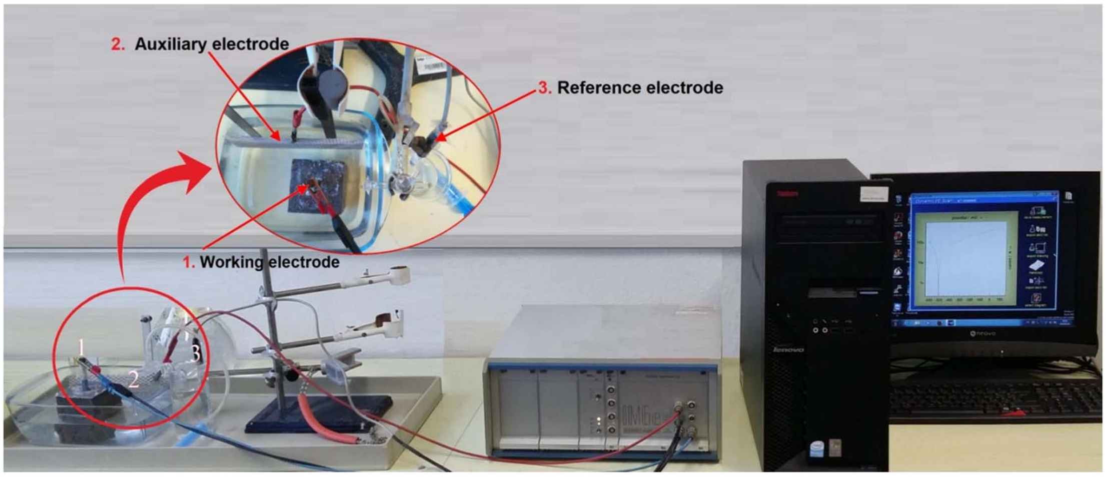

Figure 5. Tafel extrapolation circuit set up.

2.5. Chemical composition analysis

The active groups of orange peel powder, calcium nitrate, water-resisting admixture, and super-plasticiser have been detected by the FTIR spectroscopy test.

2.6. Tafel extrapolation analysis

The corrosion current has been estimated using a Tafel plot and a programmer-polarising device. The analysis was carried out on samples subjected to an accelerated corrosion process. These samples are the steel reinforcement contained in concrete cubes that had been partially immersed in the 3.5 wt% NaCl solution. Figure 5 shows the way to connect the electrodes to measure the corrosion current of reinforcing steel.

2.7. Corrosion rate

The volumetric loss of metal by unit of area and unit of time is represented by the corrosion rate (Cr) or corrosion velocity, V. It is represented in mm/year in the current suggestion, while alternative units may also be used. The corrosion rate can be calculated using the density of the metal and the corrosion current (i_{corr} or I_{corr}) expressed in $\mu A/cm^2$. For the steel, 1 $\mu A/cm^2$ corresponds to a uniform attack corrosion rate of 0.0116 mm $year^{-1}$ [52], so the corrosion rate will be:

$$C_r = 0.0116 \times i_{corr} \tag{1}$$

Where:

Cr = Corrosion rate (mm/year).

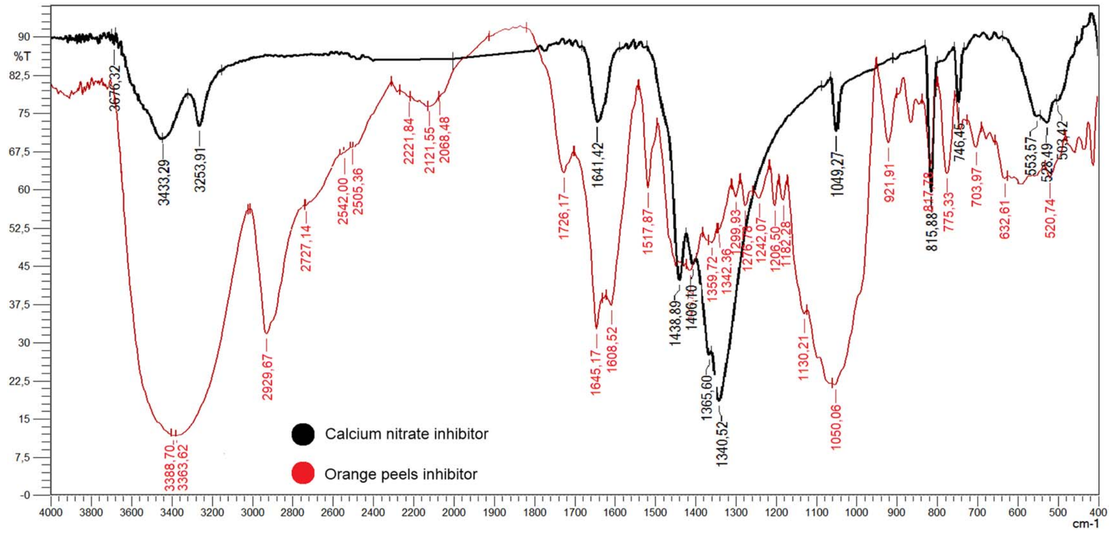

Figure 6. Fourier transform infrared spectrum (FTIR) analysis for dry orange peel powder and calcium nitrate inhibitor..

i_{corr} = Corrosion current density ($\mu A/cm^2$).

In our case, a computer-controlled potentiostat (ZAHNER type) was used to record current–potential data, which was then converted to corrosion rates using standard procedures.

2.8. Inhibitor efficiency

The efficiency of the inhibitor is measured by:

$$\text{Inhibitor Efficiency}(\%) = 100 \times \frac{(CR_{uninhibited} - CR_{inhibited})}{CR_{uninhibited}} \quad (2)$$

Where:

$CR_{\text{uninhibited}}$ = corrosion rate of the uninhibited system.

$CR_{\text{inhibited}}$ = corrosion rate of the inhibited system.

3. Results and disscusion

3.1. Chemical composition analysis of inhibitors and plasticisers

By using FTIR technology, the chemical structures of the green inhibitor (powdered orange peels) and calcium nitrate inhibitor were identified (as shown in figure 6). According to this graph, a substantial quantity of OH groups from lignin and carbohydrates are responsible for the peak at (3280.93) cm^{-1} in the high energy region of the orange peel. The distinctive band at (2925) cm^{-1} is connected to the presence of C–H stretching vibrations combined with bending vibrations around (1428) cm^{-1} of aliphatic chains (–CH$_2$– and –CH–), forming the fundamental structure of this lignocellulosic material. This peak also corresponds to the link C–O–H or C–O–R (alcohols or esters) at (1045) cm^{-1}. All of these chemical compounds have the capacity to interact with the surface of steel rebar to produce double bonds, triple ties, and aromatic ring connections that result in the production of a protective layer. Some researchers hypothesise that an increase in alkalinity near the steel reinforcement, which supports the restoration of the passivation film, and high concentrations of aluminates, which prevent chloride diffusion, are responsible for the corrosion reduction observed after adding green inhibitor to freshly reinforced concrete [53]. The OH stretching modes of the calcium nitrate active groups in figure 6 are represented by a reasonably strong absorption band at 3642 cm^{-1}. Less clarity existed in the OH stretching absorption band. The OH stretching modes also related to the wide band between 3253 cm^{-1} and 3433 cm^{-1}. The peak at 1641 cm^{-1} was created by the symmetric stretching of NO_2. C–H group deformation is shown by the wide band of 1438 cm^{-1} and 1406 cm^{-1}. The peak at 1365 cm^{-1} was induced by NO^{3-} stretching. The stretching mode of the N = symmetric O produced the peak at 1340 cm^{-1}. The NH_2 group was wagging and twisting, which matched to the strong peak at 815 cm^{-1}. The peak at 746 cm^{-1} was created by the vibration. All of these substances have the ability to interact with the surface of steel rebar to produce double bonds, triple ties, and aromatic ring connections that result in the formation of a shield surrounding it [54]. It can be seen from figure 7, which depicts the FTIR analysis for the super-plasticiser and water-resisting admixture, that the low molecular weight polyethylene glycol, with a trace amount of a lower ester, makes up the majority of the

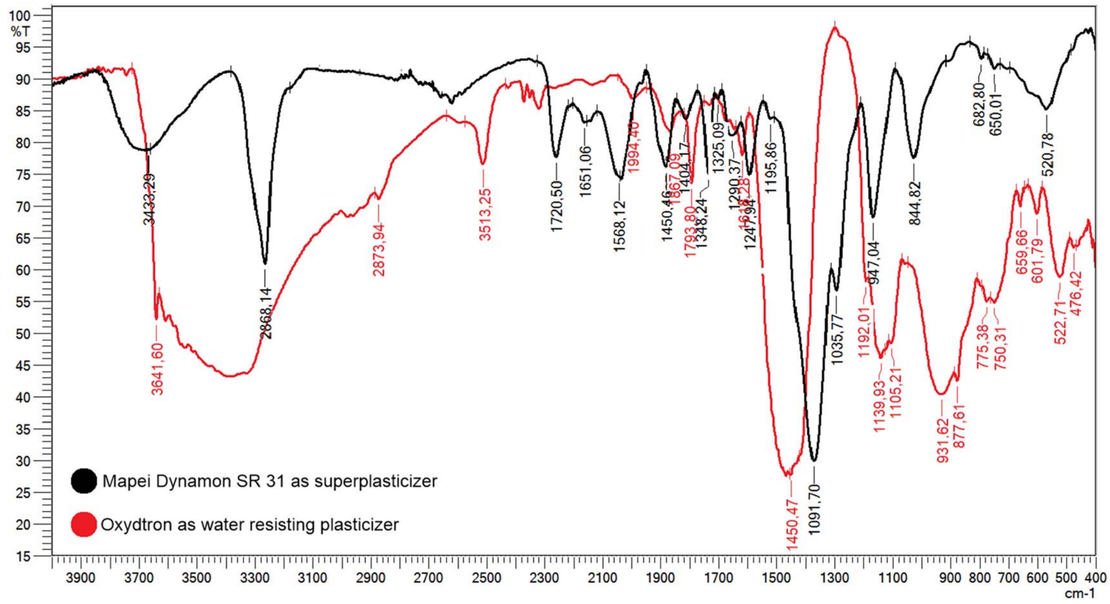

Figure 7. Fourier transform infrared spectrum (FTIR) analysis for super-plasticiser and water-resisting admixture.

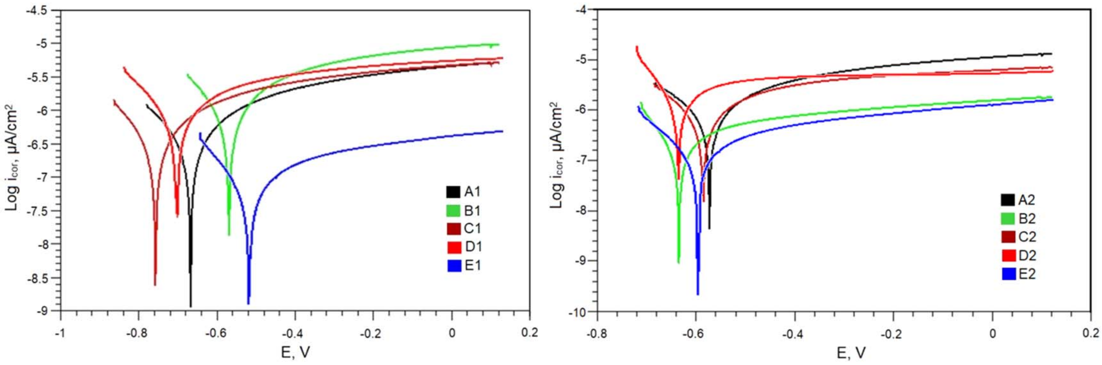

Figure 8. Polarisation curves recorded for concrete samples after immersion concrete samples in 3.5 wt % of sodium chloride solution for 18 month.

super-plasticiser. Mineral elements, including calcium oxide, silicates, and aluminates, should be present in the solid Oxydtron (nano cement), since they should be crucial to the hydration of the freshly created concrete.

3.2. Linear polarisation resistance

The electrochemical polarisation curves of reinforcing steel samples immersed in a 3.5 wt% solution of sodium chloride and implanted in concrete blocks were measured and visually shown after 18 months, as shown in figure 8. Sample C2 has been demonstrated to be the most corrosion-resistant sample while using green inhibitor. This inhibitor produced a durable protective layer surrounding the steel rebar's surface, as seen by the lower corrosion current densities in the E1 and E2 samples that included 3 wt% calcium nitrate inhibitor. The samples of A2, D2, and E2 exhibit a noticeable tendency of changing corrosion current densities in contrast to the samples of A1, D1, and E1. To increase corrosion resistance, the embedded steel rebars in the concrete blocks (A2, D2, and E2 samples) constructed with the plasticiser type water-resisting additive should undergo pozzolanic reactions.

3.3. Corrosion rate

Figure 9 illustrates how corrosion rates change with immersion time throughout the course of the 18-month test period. Rates are consistently somewhat lower when concrete samples (C1 and E1 samples) contain 3% concentrations of corrosion inhibitor combined with super-plasticiser. Figure 10 shows the findings of the corrosion rate for concrete samples with water-resisting additives. According to figure 10, corrosion rates tend

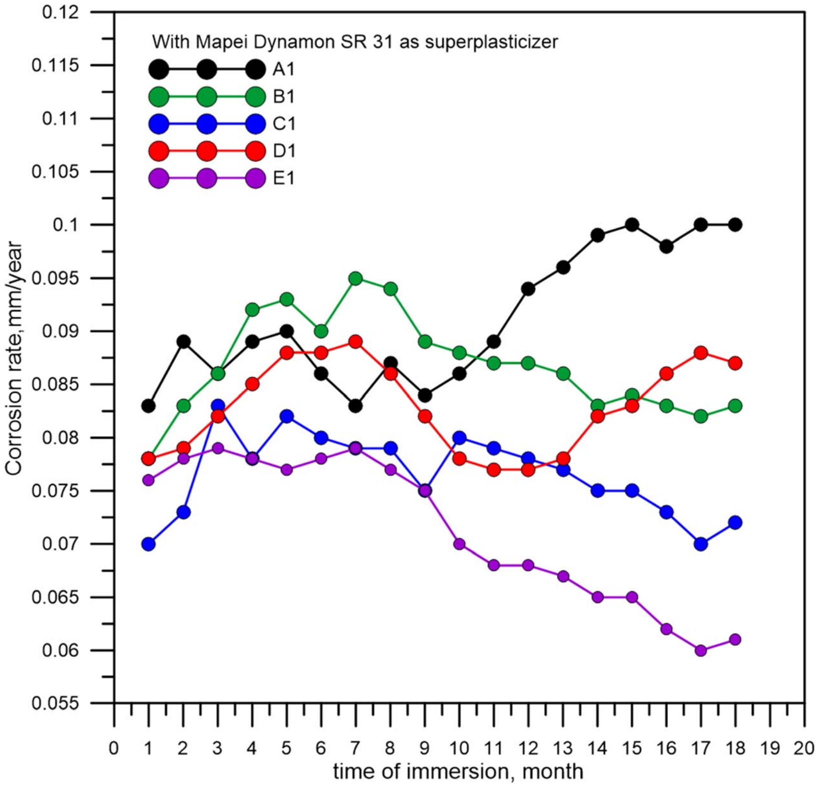

Figure 9. Corrosion rate for concrete samples with super-plasticiser after immersion for 18 months in 3.5 wt% of NaCl solution.

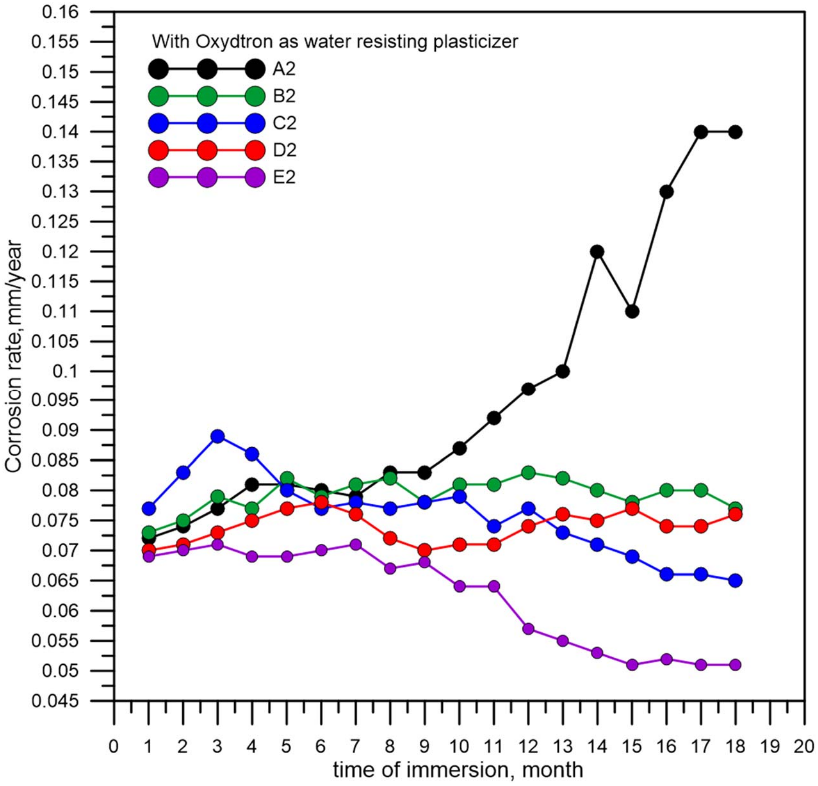

Figure 10. Corrosion rate for concrete samples with water-resisting admixture after immersion for 18 months in 3.5 wt% of NaCl solution.

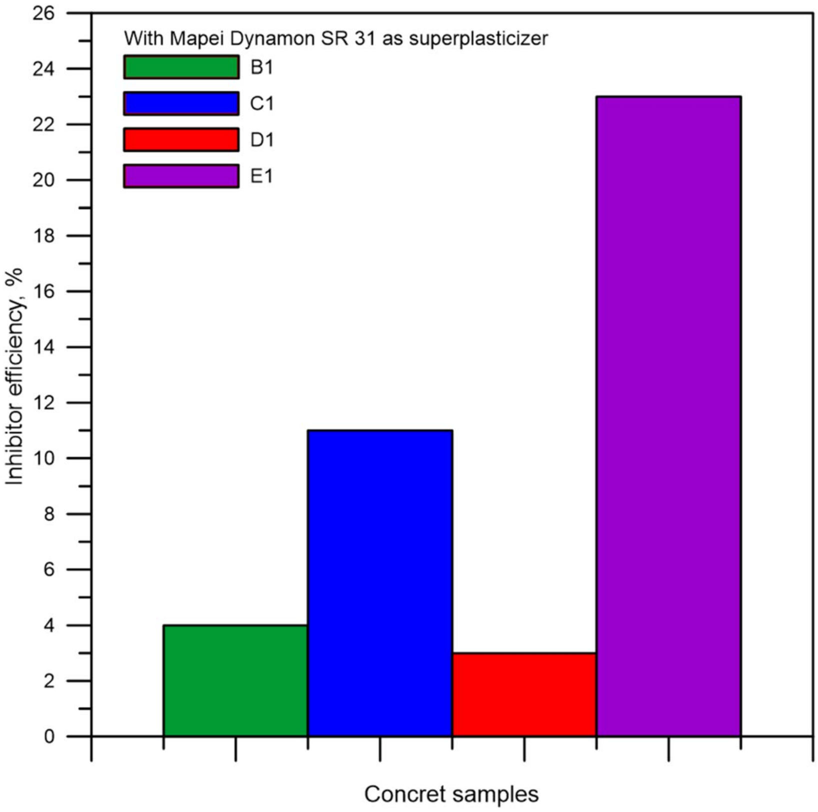

Figure 11. Inhibitors efficiency for concrete samples with super-plasticiser after 18 months of immersion in 3.5wt% of NaCl solution.

to rise with immersion time throughout the course of the 18-month test period, but they are consistently a bit lower when the samples include calcium nitrate inhibitor, particularly at 3 wt%, and when they also contain samples with a plasticiser-type water-resisting admixture. Since calcium nitrate has an effective inhibition mechanism on chloride-induced corrosion of calcium nitrate at concentrations greater than 1 wt% by weight of cement, the corrosion rate in samples E1 and E2 increased significantly over the course of the 18-month period but decreased significantly in samples with a 1% inhibitor [55]. After six months, the corrosion rate of the samples that included a green inhibitor grew less when combined with a water-resisting admixture than the corrosion rate of the samples (except for C1). This means that the green inhibitor that has been selected and tested has a highly reasonable and promising inhibitory effect, especially when paired with a plasticiser-type water-resisting admixture.

3.4. Inhibitor efficiency

The efficiency of corrosion inhibitors was calculated based on the corrosion rate diagrams listed above in figures 9 and 10 after the samples had been immersed in a 3.5 wt% NaCl aqueous solution for 18 months. In general, the efficiency of an inhibitor increases with the increase in concentration [56], which was clear for all mixes of concrete samples; the inhibitor efficiency for orange peel extract with super-plasticiser increased from 4% for a concrete mix with 1 wt% inhibitor (sample B1) to 11% for a concrete mix with 3 wt% inhibitor (sample C1). Also, in samples with calcium nitrate and super-plasticiser, the efficiency for sample D1 (with 1 wt% inhibitor) increased from 3 wt% to 23% in sample E1 (with 3 wt% inhibitor), as shown in figure 11.

In contrast, corrosion inhibitor efficiency (both types) and water-resisting admixture worked better in concrete samples than super-plasticiser due to the fact that Oxydtron plasticiser possesses more active groups and forms stronger bonds. Inhibitor efficiency with water-resisting admixture increases with inhibitor concentration; it’s clear that with a sample containing 1 wt% green inhibitor, efficiency increases from 6% to 11% at 3 wt% green inhibitor. The same thing happened with calcium nitrate and water-resisting admixture, with 1% calcium nitrate increasing by 3 wt% and 3 wt% calcium nitrate increasing by 35%, as shown in figure 12.

Calcium nitrate inhibits corrosion by promoting the formation of a passivation layer on the surface of reinforcing steel. To react with the free iron ions, the nitrate ions compete with any chloride ions present. So, in the samples E1 and E2 (3 wt% calcium nitrate and both types of plasticisers), the nitrate to chloride ion ratio at the steel level is more significant than one, causing a reaction between nitrate and iron to bind the iron into an

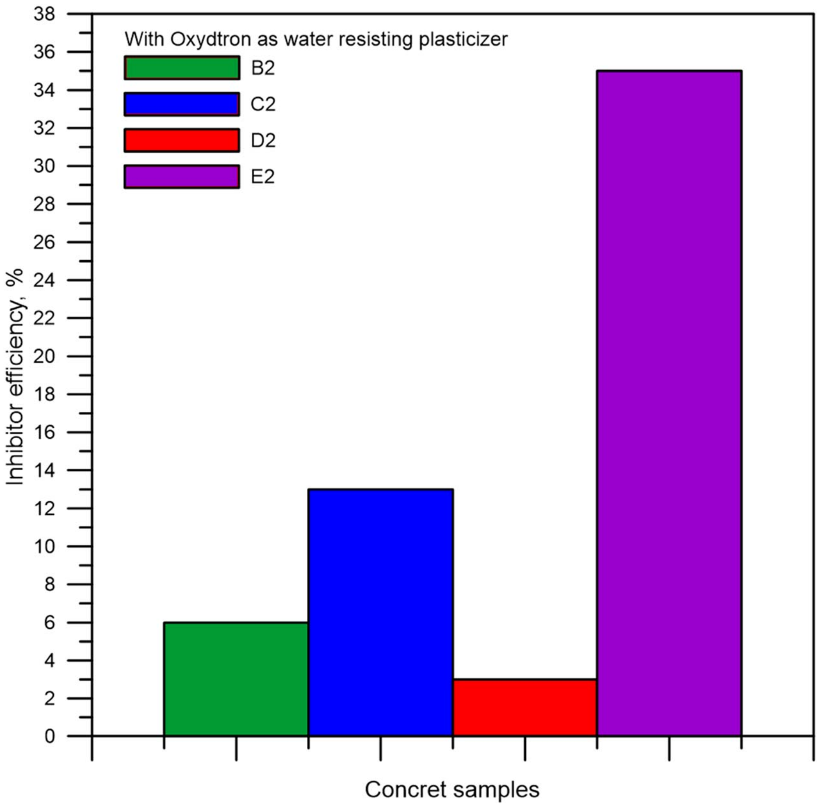

Figure 12. Inhibitors efficiency for concrete samples with water-resisting admixture after 18 months of immersion in 3.5wt% of NaCl solution.

oxide, reinforcing the passive layer on the steel. On the other hand, the ratio is less than one in samples D1 and D2 (1 wt% calcium nitrate and both types of plasticisers), which will cause the chloride ions to react with the iron to start the corrosion process. This is because the number of nitrate ions is used up during the chemical reaction between nitrate and iron.

3.5. Cost analysis

This analysis is to determine the influence of the use of orange peel extract when it is used as a green inhibitor in reinforced concrete mixtures of type XD3. The orange peels are considered a waste of Juice factories and also a waste of homes in Hungary, so in this study, waste is reduced by reuse, thus reducing the cost of producing corrosion-resistant and environment-friendly concrete. The results presented in figure 13 suggest that when a green inhibitor (orange peel extract) is added to a fresh concrete mixture; the price is practically the same compared to plain mixture. Notwithstanding, in the mixture where 1 wt% of cement was replaced with orange peel extract, it showed a decrease of almost 0.44 USD/m^3 compared with the price of reference concrete samples (A1, A2). At 3 wt% orange peel extract from the weight of cement in the mixture, it showed a decrease of almost 1.33 USD/m^3 compared to the price of reference concrete samples (A1, A2).

This study also sought to determine the impact of using calcium nitrate inhibitor as an inorganic inhibitor in reinforced concrete mixtures of type XD3. Calcium nitrate is also inexpensive and usually effective in preventing iron corrosion in the given environment. Furthermore, calcium nitrate can be thought of as a kind of replacement for the well-known nitrite inhibitor (which is also being targeted by some toxicity concerns).

The results shown in figure 13 indicate that when an inorganic inhibitor (calcium nitrate) is added to a fresh concrete mixture, the price increases significantly when compared to the reference mixture. When 1% of the cement was replaced with calcium nitrate, the mixture's price increased by nearly 1.51 USD/m^3 when compared to the price of reference concrete samples (A3, A4). At 3 wt% calcium nitrate by weight of cement in the mixture, the price increased by nearly 4.54 USD/m3 when compared to the price of reference concrete samples (A3, A4).

Environmentally, generating 1 m3 of cement paste in which 1 wt% and 3 wt% of the cement were replaced with orange peels or calcium nitrate inhibitors, respectively, prevents the release of about 4 kg and 12 kg of CO2 into the atmosphere. For both types of inhibitors, the cost of concrete samples with water-resisting admixture was lower than the cost of concrete samples with super-plasticiser, as shown in figure13. As a result, the samples

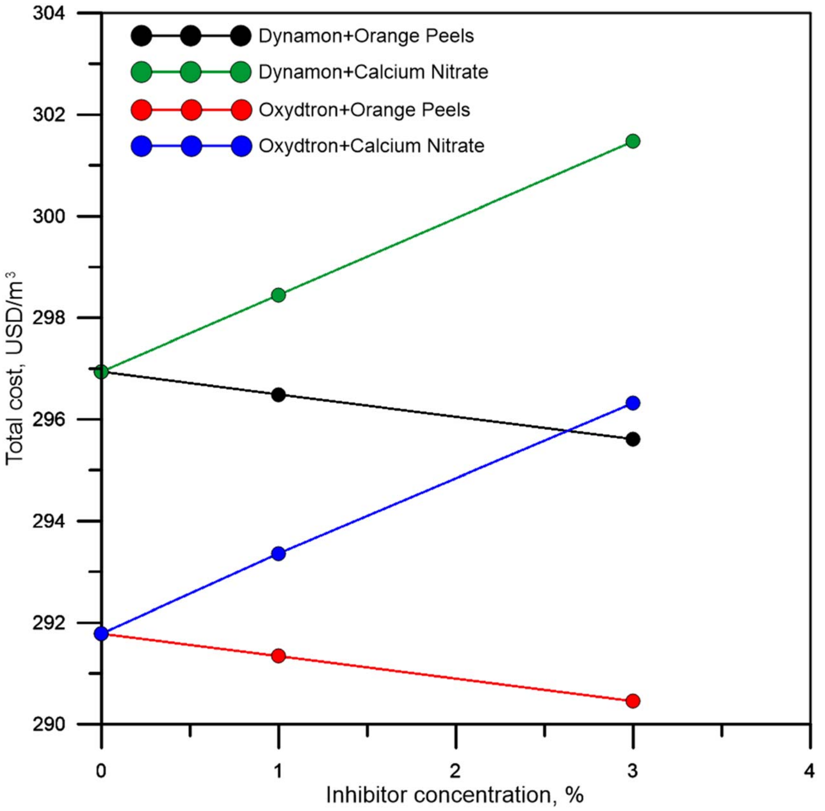

Figure 13. Cost analysis of concrete samples.

containing orange peel extract and water-resisting admixture (B2 and C2) were the least expensive when compared to the other samples.

4. Conclusion

After testing the concrete samples, we can conclude the following:

1. Adding the orange peel extract inhibitor to fresh concrete mixes demonstrated a promising positive effect on corrosion resistance with water-resistant admixture (particularly in sample C2) compared to super-plasticiser.
2. Only at a 3 wt% concentration of calcium nitrate did the concrete samples become more corrosion resistant.
3. The samples D1 and D2 received a 1wt% calcium nitrate inhibitor, but this inhibitor was not utilised, and the concrete samples' qualities were not improved.
4. The efficiency of corrosion inhibitors (both types) increases as inhibitor concentration increases, and this efficiency was higher in concrete samples containing water-resistant admixture than in samples containing super-plasticiser.
5. Using calcium nitrate at a concentration of 3 wt% resulted in a higher inhibitor efficiency of 35%.
6. The economic analysis reveals that using orange peels in the concrete mixture results in a lower price than the reference one. This inhibitor also aids in dealing with the waste problem by reducing it.

Acknowledgments

Not applicable.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Author contributions

(Shaymaa Abbas Abdulsada and Ali I. Al-Mosawi wrote the main manuscript text and prepared all the figures and tables. All authors reviewed the manuscript).

Conflicts of interest or competing interests

The authors have no competing interests as defined by Springer, or other interests that might be perceived to influence the results and/or discussion reported in this paper.

Data and code availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Supplementary information

Not Applicable.

Ethical approval

The authors certify that the ethics approval and consent to participate.

The authors certify that they NO Funding received for this work.

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